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by William G. Pritchett, Daniel M. Baechle, and Eric D. Wetzel

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**Weapons and Materials Research Directorate, ARL**

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**14. ABSTRACT**

Monofilaments with continuous, conformal metal or ceramic coatings could be used to develop new sensing and photonic technologies. This paper discusses the application of thin coatings onto meter-long monofilaments of millimeter-scale diameters. Two separate devices were designed and fabricated to produce uniform coatings over the entire surface area of a monofilament. One device enabled coatings to be applied onto multiple medium-length monofilament segments, the other onto a single longer monofilament. The magnetron sputter deposition (MSD) process was used to apply copper coatings on the order of 10–100 nanometers thick onto both nylon and polycarbonate monofilaments. The resistivity of each coating was measured, and scanning electron microscope and optical microscope analyses were performed. The process was proven viable and produced a useful product; however, a more consistent apparatus may be designed for continuous coating of monofilaments. Though only copper coatings are discussed in this report, the system could also be used to apply a variety of sputtered metal or ceramic coatings and enable new monofilament functionalities.

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sputter, monofilament, thin films, functionalization

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# 1. Introduction

A thin, uniform coating on long segments of monofilament could drastically improve the functionality of many complex fibers. Germanium-coated optical fibers could be designed to filter infrared (IR) photons.[1] This coating could be used to either protect inner signals or selectively capture exterior signals. For more complex fibers with electrically conductive cores, a thin coat of conductive copper could be selectively linked with specific cores to create an easily accessible terminal.

A length of fishing line, microtubing, or polylactic acid (PLA) coated with copper could be left to cure within an epoxy, and upon removal of the monofilament, a narrow channel with a thin outer wall of copper would remain. That channel would be open for fluid flow, and also have a conductive shell. The "vascularized" material could be used for thermal management or self-healing composites.[2]

In all of the examples discussed, the coatings are uniform around the circumference of the entire monofilament and satisfy a required thickness, on the order of nanometers. The magnetron sputter deposition (MSD) process applies relatively uniform, nanometer-thick coatings. The coatings can be made of many materials including most metals and many ceramics. The process begins by evacuating a vacuum chamber to high-vacuum conditions. Then, an inert gas such as argon is introduced, and plasma is created at the MSD source using a high voltage. The coating material target is located at the deposition source. As argon ions collide with the negatively charged target material, atoms of the target material break free. The freed atoms build up on the surface of objects placed in front of the MSD source and form thin coatings. MSD is a very effective process to create uniform, line-of-sight, nanometer-scale coatings. This paper discusses the use of MSD to create coated monofilaments and classify both their uniformity and coating thickness. A special apparatus is utilized to allow for complete line-of-sight exposure about the circumference of a filament.

# 2. System

Two apparatuses were created to coat filaments using MSD. The first apparatus, seen in figure 1, simultaneously rotated four monofilaments about their own axes. The monofilaments were attached to two sets of rotating axles. In the driven set, one of the axles was elongated and

[1] Kuriki, K.; Shapira, O.; Hart, S. D.; Benoit, G.; Kuriki, Y.; Viens, J. F.; Bayindir, M.; Joannopoulos, J. D.; Fink, Y. Hollow Multilayer Photonic Bandgap Fibers for NIR Applications. *Optics Express* **2004**, *12* (8), 1510.

[2] Esser-Kahn, A. P.; Thakre, P. R.; Dong, H.; Patrick, J. F.; Vlasko-Vlasov, V. K.; Sottos, N. R.; Moore, J. S.; White, S. R. Three-Dimensional Microvascular Fiber-Reinforced Composites. *Advanced Materials* **2011**, *23* (32), 3654.

coupled to an external motor via a rotary vacuum feed-through. Each of the four driven axles was directly connected to its neighbor by nylon gearing. In the free set of axles, there were neither gears nor couplings and each axle was free to rotate independently.

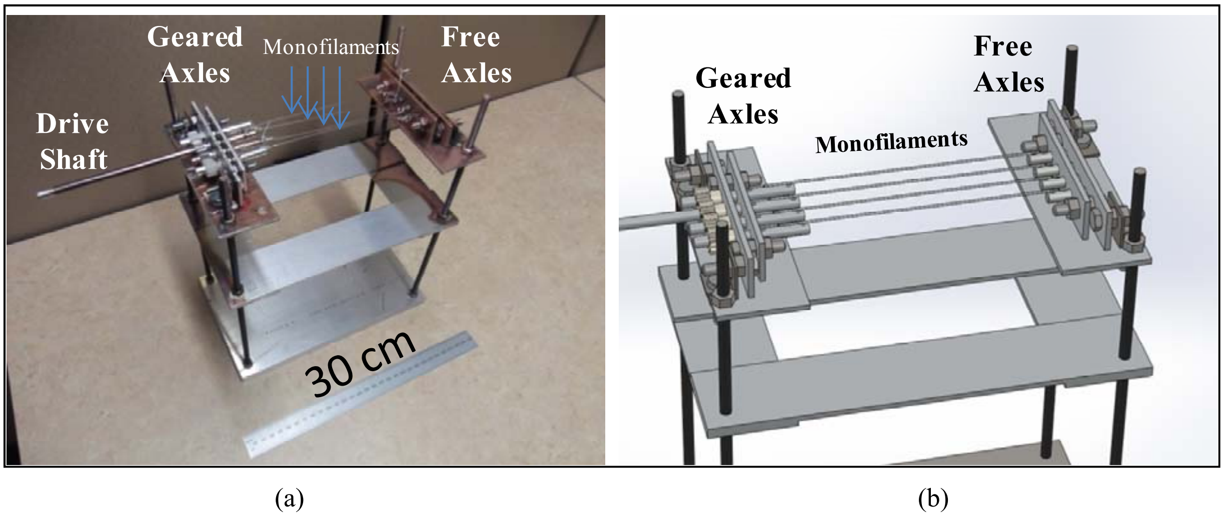


Figure 1. (a) Photograph and (b) rendering of apparatus 1 for rotating multiple monofilaments.

As the motor turned, the rotation was transferred to each of the four driven axles, which rotated each monofilament. The torsion in each monofilament was released as each opposing axle rotated freely. In the experiment, the distance between the two sets of axles was 5.25 in (13.3 cm), though that distance could be modified up to 8.25 in (21.0 cm). The majority of components in the apparatus were made of aluminum, and a select number of steel nuts, bolts, and brackets were used as well.

The second apparatus (figure 2) utilized in the experiments was designed to rotate and translate a single monofilament. The added functionality of translating the monofilament allowed for longer sections of monofilament to be coated. With the second apparatus, each point on the surface of the monofilament followed a spiral path about the longitudinal axis of the monofilament.

Two independent motors, each attached to a rotary vacuum feed-through, drove the apparatus. Each motor was coupled to a 0.625 in (15.9 mm) wide roller of Buna-N polymer. The roller surfaces were separated by 0.04 in (1 mm), and the axes of the wheels were offset by 90°. Aluminum faceplates guided the monofilament between the rollers, constraining the monofilament axis to a 45° angle with respect to each roller axis. As the monofilament passed between the two rollers, it was both rotated and translated along its longitudinal axis. The apparatus was almost completely made of aluminum with select steel, stainless steel, and Buna-N polymer components.

Both motors were driven at similar speeds, measured in revolutions per minute (RPM). After passing under the MSD source, the monofilament passed through a small hole in an aluminum plate placed 7 in (17.8 cm) from the aluminum faceplate attached to the rollers.

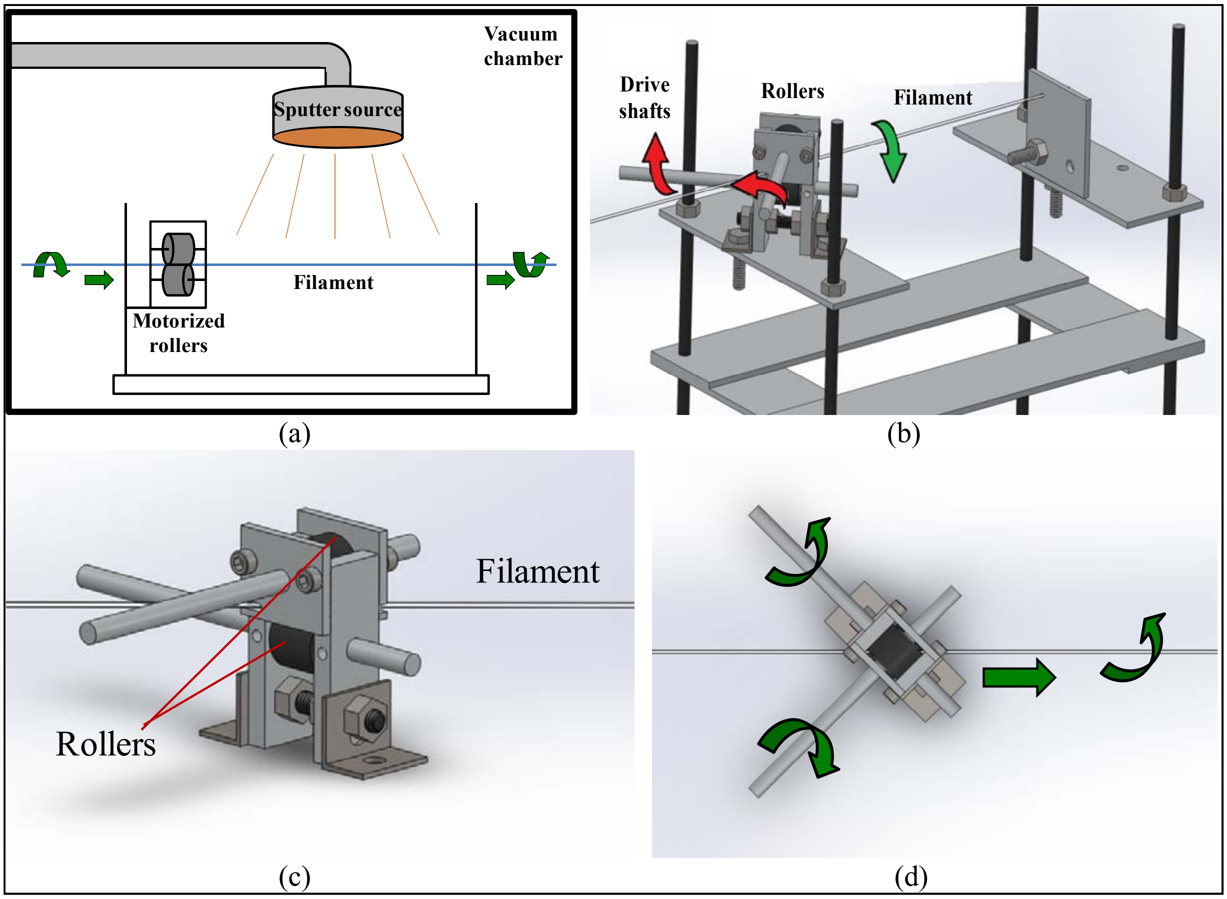


Figure 2. (a) Diagram of double-roller apparatus 2 inside vacuum chamber with MSD source; (b) rendering of double-roller apparatus 2; (c) side, and (d) top detail of roller assembly of apparatus 2.

## 3. Calculations

Though many methods exist to measure the rate of coating thickness growth on a flat surface, no procedure was found to explicitly measure the coating thickness of a cylinder. The simplest estimation was found by comparing the surface area of a flat rectangle and a cylinder, as illustrated in figure 3.

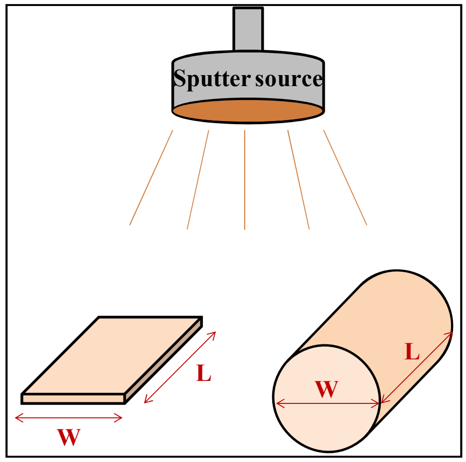


Figure 3. Illustration of geometry used for coating rate calculations.

$$\frac{Surface\ Area\ of\ a\ Rectangle}{Surface\ Area\ of\ a\ Monofilament\ Length} = \frac{W \cdot L}{\pi \cdot W \cdot L} = \frac{1}{\pi} \tag{1}$$

Equation 1 suggests that the rate of coating growth on a length of rotating monofilament is $\frac{1}{\pi}$ times that of a flat surface.

Another method of measuring coating thickness on a cylindrical body utilizes the measurement of electrical resistance (*R*), a relatively simple measurement. The resistance of any object is dependent on the resistivity of the material (*ρ*), the length of the object (*L*), and the area (*A*) through which electric current passes.

$$R = \rho \frac{L}{A} \tag{2}$$

The resistivity of copper is 18 orders of magnitude smaller than the resistivity of the monofilament material. Therefore, practically all of the current flows through the copper coating and the area through which electric current passes can be defined as the cross-sectional area of the coating. The cross-sectional area of the coating is dependent on the diameter of the monofilament (*D*) and the coating thickness (*t*).

$$\begin{aligned} A_{coating} &= A_{filament+coating} - A_{filament} \\ A_c &= \pi\left(\frac{D}{2}+t\right)^2 - \pi\left(\frac{D}{2}\right)^2 \\ A_c &= \pi\left(D+t\right)t \approx \pi D t \end{aligned} \tag{3}$$

In this calculation, an approximation is made for simplification, because the coating thickness (t) is six orders of magnitude smaller than the monofilament diameter. Combining equations 2 and 3, the resistance over a specified length of monofilament is inversely proportional to the coating thickness.

$$R = \rho \frac{L}{\pi D} \cdot \frac{1}{t} \tag{4}$$

The coating thickness is dependent on the residence time ($T$) in which the MSD process occurs and the rate at which the coating grows ($s$). The growth rate is dependent on the power applied to the MSD source and the distance between the sample and the MSD source.

$$t = sT \tag{5}$$

For apparatus 1, which only rotates the filaments, the residence time can be assumed to be equal to the overall deposition time if the rotation speed is sufficiently high and the deposition time is sufficiently long. For apparatus 2, the residence time cannot be explicitly measured, but can be calculated. Each section of monofilament is only exposed to free sputtered atoms in a limited gap. The time an infinitesimally small section of monofilament spends in that gap is dependent on the length of the gap ($l$), and the linear velocity of the monofilament ($v$).

$$T = \frac{l}{v} \tag{6}$$

The velocity at which the monofilament translates is dependent on the angular velocity of the rollers ($\omega$), the diameter of the rollers ($d$), and the angle at which the monofilament crosses each roller ($\alpha$).

$$v = \omega \frac{d}{2} \sin(\alpha) \tag{7}$$

If the axis of the monofilament were perpendicular to the axes of both rollers, the linear velocity of the monofilament would be equal to the linear velocity of any point in contact with the monofilament, assuming the monofilament does not slip on the rollers. However, if the axis of the monofilament were parallel to the two roller axes, there would be no translation of the monofilament and it would simply be rotated about its longitudinal axis.

Putting together equations 5, 6, and 7, the coating thickness is inversely proportional to the angular velocity of the rollers.

$$t = \frac{2sl}{d \sin(\alpha)} \cdot \frac{1}{\omega} \tag{8}$$

Finally, equations 4 and 8 combine to show the resistance measured is linearly proportional to the angular velocity of the drive rollers.

$$R = \rho \frac{Ld \sin(\alpha)}{2\pi l D s} \cdot \omega \tag{9}$$

## 4. Experiment Set 1

To test uniformity and coating thicknesses, a set of simple experiments were performed on 6-in (15 cm) long monofilament segments. Three types of monofilaments were utilized in this experiment set: a commercially available nylon fishing line with a diameter of 0.028 in (0.7 mm), a larger complex polycarbonate fiber with a diameter of 0.06 in (1.4 mm), and a PLA monofilament with a diameter of 0.02 in (0.5 mm). Apparatus 1 was used in this experiment set, coating two monofilament segments at a time. Copper coatings were applied using 120–150 W of direct current (DC) power for time periods of 2–4 min. In each test the segments were rotated at 3–4.5 RPM. The segments were mounted 6 in (15 cm) below the deposition source.

The MSD process took place inside a 3.85-$ft^3$ (0.11 $m^3$) vacuum chamber. A commercially manufactured MSD source (Onyx-3 ICSTD, Angstrom Sciences Inc.) was used in the experiment. The chamber was evacuated below $5.0 \cdot 10^{-6}$ Torr using a rough pump (DUO10M, Pfeiffer) and turbomolecular pump (TMU521P, Pfeiffer). Then, argon was added into the chamber to reach a working pressure of 1.0 mTorr. The monofilaments were rotated at a constant rate, and the high voltage was applied to the 75-mm diameter, 7-mm-thick 99.99% pure copper target (EJTCUXX403A4, Kurt J. Lesker Co.) for specified times.

## 5. Experiment Set 2

A second experiment was performed to incorporate both rotation and translation of the monofilament, yielding longer sections of coated monofilament. A 37-in (94 cm) polycarbonate monofilament with a 0.04-in (1 mm) diameter was tested in this experiment. Deposition was again performed with a copper MSD target. The monofilaments were mounted 6 in (15 cm) from the MSD source. Deposition occurred for 5–20 min in each trial.

The MSD process was performed in a similar manner to experiment set 1; however, apparatus 2 was utilized. Once the specified power was applied to the MSD source, creating plasma, the monofilament was rotated and translated through the flow of freed copper atoms leaving the MSD source. In these experiments, the rate at which the monofilament spooled past the MSD source was varied between trials. In one trial, the power applied to the MSD source was altered as well.

The entire length of each monofilament was not fully coated. Sections at the beginning and end were left uncoated to aid in guiding the monofilament along the walls of the vacuum chamber and through the rollers. Also, the first and last 7 in (18 cm) of monofilament exposed to the

MSD source were only partially coated because they were only exposed to the MSD source for a portion of the intended residence time. However, there was a significant section near the middle of each monofilament that was uniformly coated. If a longer monofilament were coated, the fully coated portion of the monofilament could be extended, without altering the lengths of the partially coated and uncoated sections.

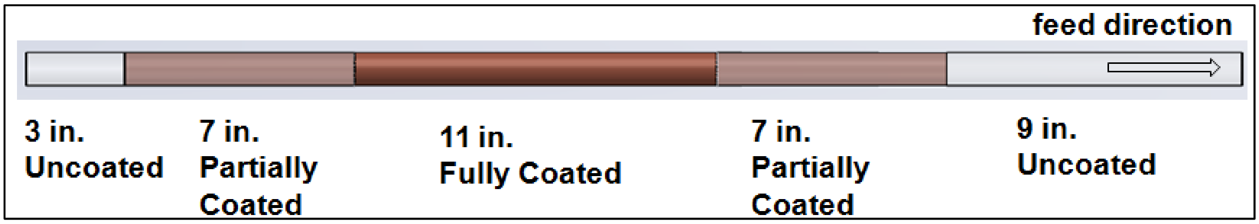


Figure 4. Coating layout of a 37 in (94 cm) coated monofilament.

## 6. Characterization

Small witness strips of silicon were placed 0.4 in (1 cm) below the monofilament and later tested with Rutherford Backscattering Spectroscopy to measure the thickness of coating accrued on a flat surface over the course of a trial. This measurement was used to calculate the rate of coating deposited on a flat surface per minute.

The coatings on the monofilaments were first characterized by measuring the resistance across a 10- or 16-cm segment with an inductance-capacitance-resistance (LCR) meter (model 879, BK Precision). As discussed in the calculations section of this paper, the resistance measured should be inversely proportional to the copper coating thickness.


Figure 5. Measuring coating resistance with an LCR meter.

Silver (Ag) paint was applied at contact points on the monofilament, either 10 or 16 cm apart. Once the paint was dry, the alligator clips of the LCR meter were attached to the monofilament at the Ag-painted contact points. The monofilaments were also characterized by imaging using a Scanning Electron Microscope (SEM) and an optical microscope.

## 7. Results

Experiment 1 yielded five measurable data points, found in table 1. In table 1, coating thickness is calculated from measured resistance using equation 4 above. Copper coatings were successfully applied to three types of filaments, each with a distinct material and diameter. Each coated monofilament was conductive and completely coated. A coated and uncoated filament from experiment set 1 can be seen in figure 6.

Table 1. Experiment set 1 parameters and results.

| Rotation Rate (RPM) | Filament Diameter (mm) | Filament Material | MSD Power (W) | Residence Time (min) | Measurement Distance (cm) | Resistance (Ω) | Coating Thickness (nm) |
|---|---|---|---|---|---|---|---|
| 3 | 1.4 | Polycarbonate | 150 | 6 | 10 | 73.5 | 5.2 |
| 3 | 0.7 | Nylon | 150 | 4 | 10 | 77.0 | 9.6 |
| 4.5 | 0.5 | PLA | 100 | 5 | 16 | 745.0 | 2.3 |
| 4.5 | 0.5 | PLA | 100 | 10 | 16 | 224.0 | 7.6 |
| 4.5 | 0.5 | PLA | 100 | 10 | 16 | 189.0 | 9.0 |

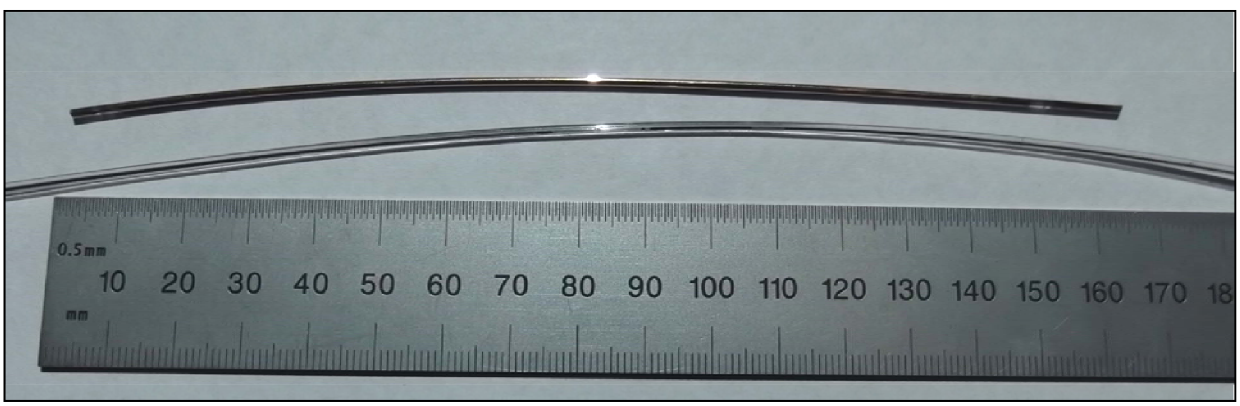


Figure 6. Coated (top) and uncoated (bottom) 1.4-mm diameter monofilament.

Results of experiment set 2 are summarized in table 2. In table 2, coating thickness is calculated from measured resistance using equation 4 above. A long, coated filament from experiment set 2 can be seen in figure 7. The data collected in experiment set 2 demonstrate a definitive positive correlation between the rotation rate of the drive wheels and the resistance measured through the coating of each monofilament. The data follow a linear fit with an $R^2$ value of 0.9748, as seen in figure 8. The coating in each segment measured was conductive.

Table 2. Measured resistance of seven coated monofilaments utilizing apparatus 2.

| Sample | Roller (RPM) | Filament Feed Rate (cm/min) | Residence Time (min) | Resistance Over 10 cm (Ω) | Coating Thickness (nm) |
|---|---|---|---|---|---|
| 1 | 0.66 | 2.7 | 6.66 | 24.3 | 22.1 |
| 2 | 0.66 | 2.7 | 6.53 | 24.3 | 22.0 |
| 3 | 1 | 4.0 | 4.48 | 80.0 | 6.7 |
| 4 | 1.1 | 4.4 | 4.00 | 119.4 | 4.5 |
| 5 | 1.1 | 4.5 | 3.94 | 90.4 | 5.9 |
| 6 | 2 | 8.1 | 2.18 | 323.8 | 1.7 |
| 7 | 2 | 8.6 | 2.06 | 345.8 | 1.6 |

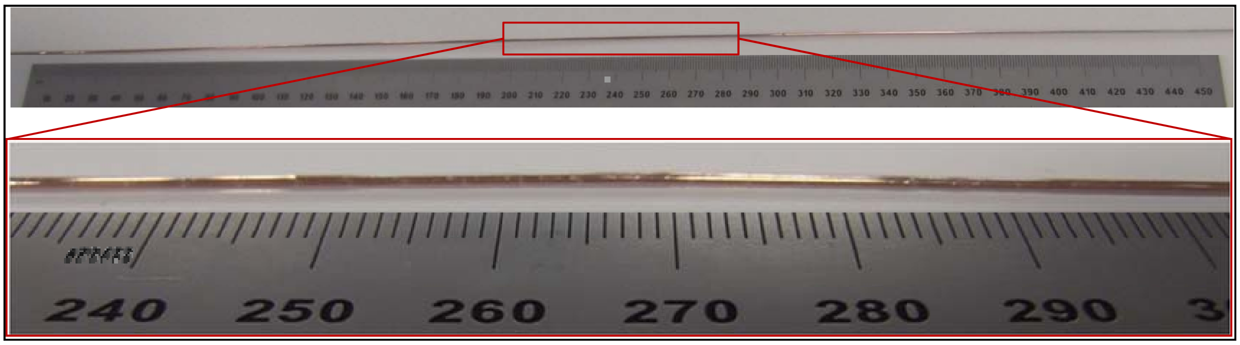


Figure 7. 18-in (46 cm) segment of coated monofilament.

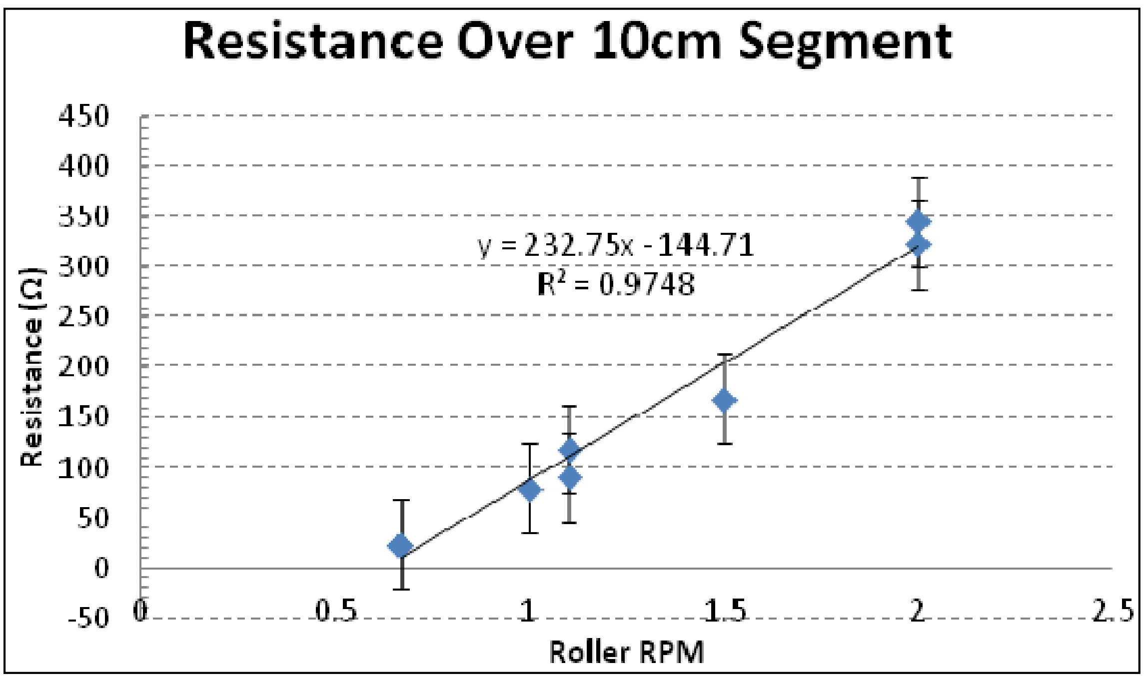


Figure 8. Measured resistance of seven coated monofilaments utilizing apparatus 2.

The calculated coating thickness data is similar to the measured resistance data. The trend in figure 9 supports the theoretical calculations with an inverse power relation between the coating thickness and roller RPM. However, the asymptote is found at a nonzero roller RPM.

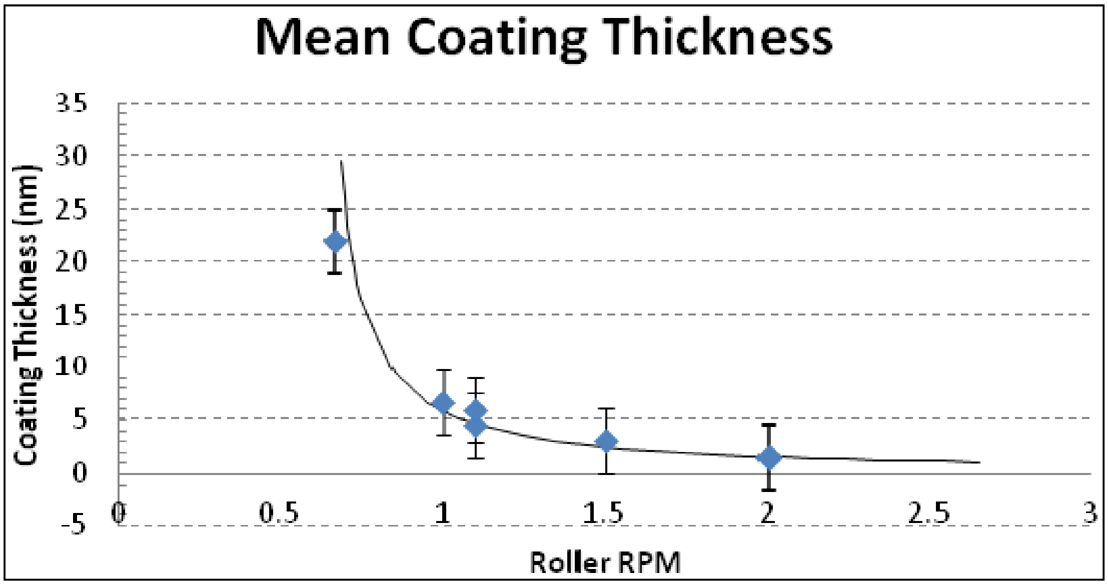


Figure 9. Calculated mean coating thickness of seven coated monofilaments using apparatus 2.

Microscopic dents and imperfections were present in the unprocessed polycarbonate monofilament. Many more lines, marks, and dents were observed in the monofilament surface after passing through apparatus 2, including a prominent line following a spiral path (figure 10). In coated monofilaments, the coating was extremely conformal and imperfections similar to the uncoated monofilament were observed. No large gaps or holes in the copper coating were observed.

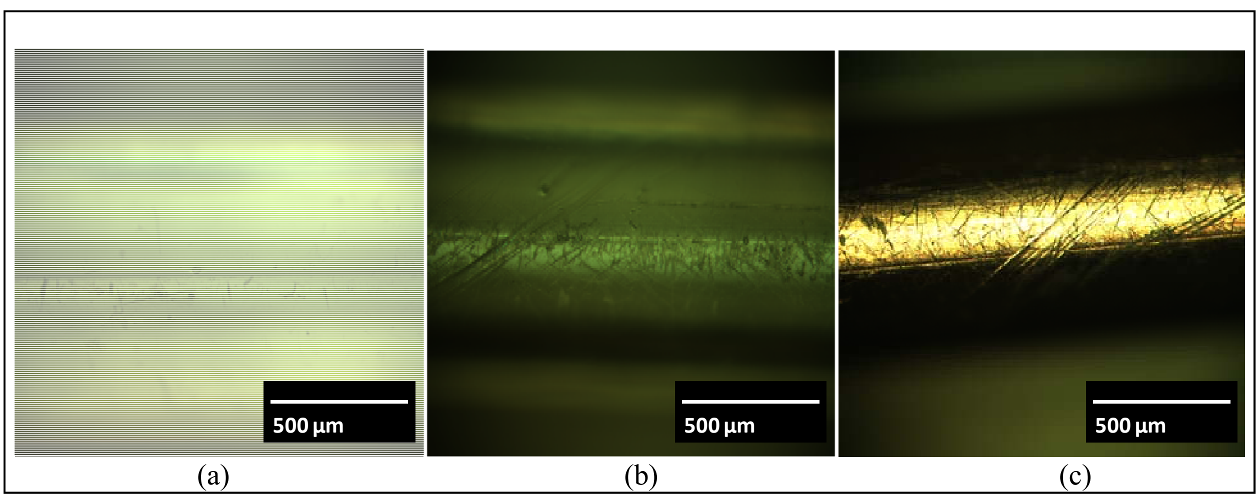


Figure 10. 10× optical micrographs of (a) unprocessed polycarbonate monofilament, (b) polycarbonate monofilament passed through apparatus 2 without coating, and (c) copper coated polycarbonate monofilament (sample 6).

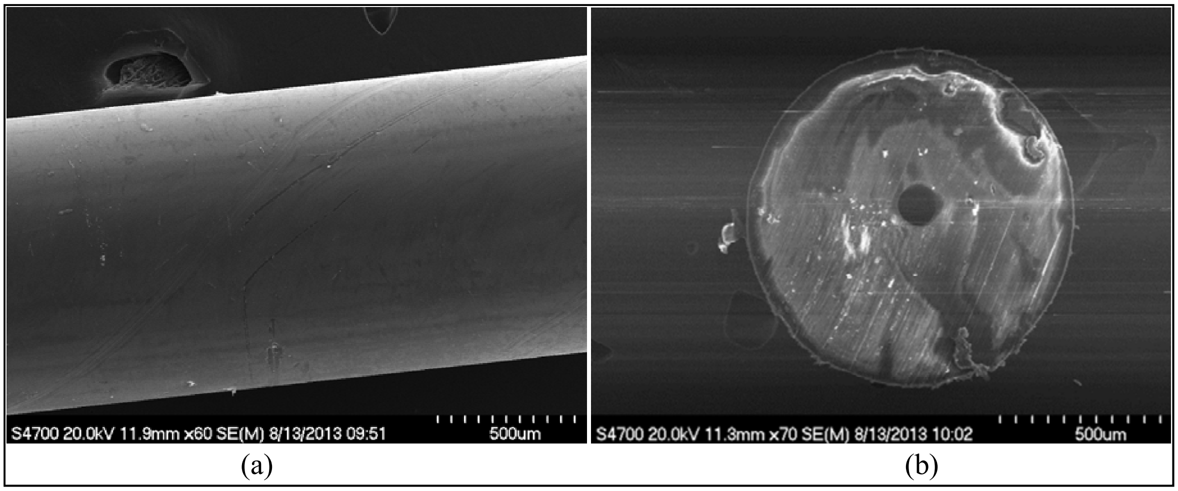


Figure 11. SEM images of copper coated polycarbonate monofilament with center channel. (a) Profile of sample 6 and (b) cross-sectional view of sample 1.

Using SEM, the copper coated monofilament was observed to be smooth with little to no indications of flaking or cracked coating (figure 11). Uniform coating was observed about the entire circumference of the monofilament.

## 8. Discussion

After concluding experiment set 1, very few conclusions could be made with respect to the coating growth rate on a rotating monofilament. However, it was conclusive that coatings applied were conductive and the resistance of each coating could be easily measured with the use of Ag paint and an LCR meter.

The results of experiment set 2 support our hypothesis that the slower a monofilament is fed through the MSD process, the less resistive the coating will be. The lower resistance corresponds to the presence of a thicker coating of copper. The consistent conductivity of each coating suggests a uniform coating process with thorough coverage.

The strong positive correlation and linear trend of figure 8 support the theoretical thickness calculations. However, it is inconclusive why the magnitude of the y-intercept of the linear fit line in figure 8 is so large. Theoretically, the linear fit should pass through the origin. As the linear velocity of the monofilament approaches zero, an infinitely thick coating would be created, which would have infinite conductivity. Simply, an infinite coating thickness would have no resistance. However, the linear fit on the data collected suggests an infinitely thick coating is achievable below 0.6125 RPM. More thorough data collection will aid in verifying a reliable value for the relation between the motor RPM and the coating resistance. However, it is conclusive that there is a strong, linear, positive correlation between the two.

The increase in microscopic markings observed on the surface of the monofilaments after passing through the rollers of the second apparatus could have a negative impact on monofilament performance in applications where a microscopically smooth surface is critical. However, the conformity of the coating and consistent conductivity observed suggest the alteration of surface texture will have little impact in most applications. The conformity of the coating observed also suggests that a uniform coating could be applied to even more sophisticated surface architectures.

## 9. Conclusions

These two experiments provide consistent data to suggest proof-of-concept and possibilities for future research. Either device may be used to coat monofilaments with the MSD process. The apparatus from experiment set 1 can be used to coat multiple filaments up to 7 in (94 cm) long. The diameter of the monofilament is relatively unrestrained with the maximum tested being 3 mm. The monofilaments can be either rigid or flexible.

The apparatus from experiment set 2 can coat much longer segments of monofilament. However, the characteristics of the monofilaments which can be coated are limited. Due to the limited space within the vacuum chamber, the monofilament must be moderately flexible to bend as it strikes the chamber wall. The monofilament must also be moderately stiff so that it remains straight while it is exposed to the MSD source. The diameter of monofilament is constrained to about 0.5–2.0 mm. However, the stiffness of the monofilament is more crucial than its diameter.

## 10. Future Work

Utilizing the two devices, many monofilament and coating combinations can be designed and fabricated. PLA monofilaments can be coated with 10–20 nm of copper to be used in electrical treeing experiments. Microtubing can also be coated to test the possibility of replacing sacrificial PLA monofilaments with reusable microtubes.

Multiple additions, refinements, or new apparatuses could be designed to improve consistency and versatility of this process as well. Performing this process within a larger vacuum chamber would allow for less restricted movement of the monofilament before and after passing by the MSD source. In experiment set 2, the restraints imposed by the walls of the vacuum chamber greatly affected the length and types of monofilaments that could be coated using apparatus 2. Utilizing a larger chamber would increase the amount of time required to pump down to high-vacuum conditions; however, it may also increase the length of monofilament that can be coated by an order of magnitude or more.

A motorized spooling mechanism, which would gather the coated monofilament after passing the MSD source, may be used in conjunction with apparatus 2 to control the translation of more flexible monofilaments.

Another option is to design a new apparatus with two motorized spools. Each spool would rotate about its own axle, either taking up or letting out monofilament. Simultaneously, the two spools would rotate together about the axis of the monofilament. Therefore, a flexible monofilament could be rotated about its axis as it is transferred from one spool to the other.

Many thousands of combinations of monofilaments and coatings may be created utilizing these devices. All that is required is the design and testing of each combination.

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## List of Symbols, Abbreviations, and Acronyms

| | |
|---|---|
| $A$ | area (units, $mm^2$) |
| Ag | silver |
| $d$ | roller diameter (units, mm) |
| $D$ | monofilament diameter (units, mm) |
| DC | direct current |
| in | inch |
| IR | infrared |
| $l$ | monofilament exposure gap (units, cm) |
| $L$ | length (units, cm) |
| LCR | inductance, capacitance, resistance |
| min | minute |
| MSD | magnetron sputter deposition |
| PLA | polylactic acid |
| RPM | revolutions per minute |
| $R$ | resistance (units, $\Omega$) |
| $s$ | coating rate (nm/min) |
| SEM | scanning electron microscope |
| $t$ | coating thickness (units, nm) |
| $T$ | time (units, s) |
| $\rho$ | density (units, $g/cm^3$) |
| $v$ | monofilament linear velocity (units, cm/min) |
| $\omega$ | roller angular velocity (units, rad/s) |
| $\alpha$ | roller-monofilament angle |

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